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Enhanced Heteroepitaxy

30 June 1978

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Lincoln Laboratory

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

LEXINGTON, MASSACHUSETTS

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This technical report has been reviewed and is approved for publication.

FOR THE COMMANDER

Raymond L. Loiselle

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MASSACHUSETTS INSTITUTE OF TECHNOLOGY
LINCOLN LABORATORY

ENHANCED HETEROEPITAXY

SEMIANNUAL TECHNICAL SUMMARY REPORT
TO THE
DEFENSE ADVANCED RESEARCH PROJECTS AGENCY

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LEXINGTON MASSACHUSETTS

ABSTRACT

Progress in research on a new method for controlling the crystallographic orientation of overlayer films using submicrometer-resolution surface-relief structures is reported. Abstracts of two theses on topographical control of overlayer orientation are included. One deals with orientation of solid crystals, while the other deals with orientation of liquid crystals.

CONTENTS

INTRODUCTION

The objectives of this research program during calendar year 1978 are to:

(1) Determine the feasibility of using submicrometer-dimension surface-relief structures to control the orientation of a variety of deposited thin films,

(2) Determine if single-crystal films of low defect density can be produced using artificial surface-relief structures,

(3) Determine if device quality AlN or ZnO films can be produced on SiO_2 or Si_3N_4 over Si, and

(4) Determine if single-crystal silicon can be produced on SiO_2 or Si_3N_4 either as a continuous film or in certain localized regions, with the long-range objective of permitting three-dimensional integration of silicon devices.

The tasks within this program include: (1) the development of a technology for fabricating the required submicrometer surface-relief structures, (2) the deposition of thin film material, and (3) the analysis of structures fabricated and their influence on thin film growth and orientation.

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ACCOMPLISHMENTS

During the first half of 1978, the Ph.D. thesis of Dale C. Flanders and the M.S. thesis of David C. Shaver were submitted and accepted by the Department of Electrical Engineering and Computer Science at M.I.T. The former included a theoretical analysis of the influence of artificial surface-relief structures on the orientation of solid- and liquid-crystalline overlayers, a detailed description of relief-structure fabrication techniques, and experimental results on alignment of tin, KCl, and the liquid crystals MBBA and M24. The latter thesis extended the work on alignment of liquid crystals using surface microstructures, and demonstrated a liquid-crystal display based on the use of metal gratings to both align liquid crystals and polarize light. Abstracts of both these theses are included in this report as Appendices A and B, respectively. Edited versions of the two theses will be submitted as Technical Reports under this contract.

In addition to the work reported in the above theses, accomplishments during the first half of 1978 included the following:

(1) Demonstration of large-grain (~200-μm) regrowth of amorphous and small-grain polycrystalline silicon on SiO_2 using Ar ion laser annealing.

(2) Demonstration that silicon crystallites grown by the CVD process

$$SiH_2Cl_2 \xrightleftharpoons{H_2 + HCl} Si + 2HCl$$

appear to have a weak tendency to align relative to the edges of 3.8-μm period surface-relief gratings etched into Si_3N_4.

(3) Development of a mathematical model for the grating-doubling process. This process permits gratings of spatial period d/2 to be exposed using master gratings of period d. By this process, using soft x-ray radiation, it should be possible to expose spatial periods of about 200 Å.

(4) Demonstration of the grating-doubling process by exposing a 2000-Å-period grating from a 4000-Å-period master.

(5) Development of a process for producing saw-tooth gratings with facets at the intersection angle of (111) planes (109°).

(6) Development of a technique for preparing samples so that relief structures, and the early stages of film growth, can be inspected by transmission electron microscopy.

(7) Demonstration, by transmission electron microscope analysis, that sidewalls of relief structures produced by reaction ion etching are within 11° of the vertical and have corner radii or curvature less than 50 Å.

APPENDIX A

ORIENTATION OF CRYSTALLINE OVERLAYERS ON AMORPHOUS SUBSTRATES BY ARTIFICIALLY PRODUCED SURFACE RELIEF STRUCTURES

By

Dale Clifton Flanders

Submitted to the

Department of Electrical Engineering and Computer Science

on January 20, 1978 in partial fulfillment of the
requirements for the Degree of Doctor of Philosophy

ABSTRACT

Overlayers of crystalline materials on smooth amorphous substrates tend to be more or less random polycrystalline. The absence of long-range order in the amorphous substrate is reflected in the absence of long-range order in the overlayer. The new concept investigated in this work is that a single crystal film can be produced on an amorphous substrate by introducing an artificial surface relief structure having long range order. A simple thermodynamic argument indicates that these surface relief structures need not have dimensions of the order of the lattice parameter of the crystalline overlayer but rather structures can be used whose dimensions are comparable to the size of the naturally occurring single crystal grains of polycrystalline films. It is argued that at equilibrium an overlayer material which exhibits an anisotropic interfacial tension (this includes the liquid crystal mesophases as well as solid crystals) will adopt a unique single-crystal orientation with respect to a suitable surface relief structure on an amorphous substrate. For example, it is shown that at equilibrium a cubic material whose $\{100\}$ planes have minimum interfacial tension will be oriented with $\{100\}$ parallel to a substrate, and a $\langle 100 \rangle$ direction parallel to the groove direction of a square-wave grating on the substrate.

It was found that the major problem in experimentally demonstrating the predicted orientation effects was the fabrication of the required surface relief structure. New very soft X-ray lithographic and reactive-ion-etching fabrication techniques were developed. With these techniques 160 nm linewidth square-wave gratings having smooth vertical sidewalls and sharp corners with less than 5 nm curvature were fabricated in amorphous silicon dioxide.

A model of nematic and smectic A liquid crystals indicates that simple square-wave structures should induce uniform "single crystal" orientation of these materials. Experiments were performed using the liquid crystals MBBA and M-24. As expected, uniform orientation was induced in MBBA in the nematic phase and in M-24 in the nematic and smectic A phases.

A detailed model of the (nonequilibrium) thin film growth process showed that under certain deposition conditions a surface relief structure could induce a solid crystalline deposit to acquire the single crystal orientation predicted by the equilibrium interfacial tension model. Experiments were performed using square-wave grating structures on amorphous SiO_2 substrates. Depositions of potassium chloride from aqueous solution and tin by vacuum evaporation were done on these structures. Potassium chloride crystallites were oriented with $\{100\}$ parallel to the substrate and $\langle 100 \rangle$ parallel to the groove direction as predicted by the thin film

growth model. The orientation effect was not observed on structures whose square profile had been rounded. This is explained qualitatively by the model of thin film growth. A series of tin depositions on square-wave gratings yielded results consistent with the model of thin film growth, but a strong orientation effect was not observed. Only weakly preferred orientation seems to have been induced by the surface relief structure. It is concluded that smaller periodicity grating structures with sharper edges and corners will be required to induce a strong orientation effect with tin.

A new method of orienting crystalline (anisotropic) overlayers on an amorphous substrate by surface relief structures on the substrate has been analyzed and demonstrated. New submicrometer fabrication techniques had to be developed in order to demonstrate the orientation effect. These techniques may have broad application in the fields of microelectronics and integrated optics as well as in the work presented here. It is believed that the models and demonstrations of overlayer orientation presented here are but a first step in what will be an exciting new field of investigation. In essence, a new degree of freedom has been introduced in the science and technology of surfaces and thin film growth.

Thesis Supervisor: Dr. Henry I. Smith

Title: Adjunct Professor of Electrical Engineering and Computer Science and Assistant Group Leader at M.I.T. Lincoln Laboratory.

APPENDIX B

THE ALIGNMENT OF LIQUID CRYSTALS BY SURFACE GRATINGS

by
David Carl Shaver
Submitted to the Department of Electrical Engineering
and Computer Science
on June 6, 1978 in partial fulfillment of the
requirements for the Degree of Master of Science

ABSTRACT

Square-wave grating structures with periodicities ranging from 3200 Å to 12 μm were etched into fused quartz substrates, and the effect of such gratings on liquid crystal alignment was studied. Gratings with periodicities below 4 μm appear to be required to align typical room temperature nematic liquid crystals. At larger periodicities a pronounced defect texture forms. The defect texture is created during nucleation and growth of the nematic phase as it cools from the isotropic phase. The defect texture is stabilized by adsorption of an oriented molecular layer on the substrate surfaces. This adsorbed layer exerts an orienting torque on the bulk liquid crystal. Experiments were performed to demonstrate the existence of such an adsorbed layer.

The tilt angle of the nematic director from the plane of quartz substrates was measured for liquid crystals used in the alignment experiments. M24 and the 'heptyl/butyl mixture' align with the director in the substrate plane. MBBA aligns with a tilt angle of about 23 degrees on fused quartz, whether or not a grating structure is present.

Surface gratings were also formed by patterning a monolayer of DMOAP. Such patterned organic monolayers, which have no appreciable surface relief, are effective at aligning liquid crystals. This represents a new approach to liquid crystal alignment.

High quality alignment of the smectic A phase of M24 was induced by a 3200 Å period square-wave surface relief grating.

A novel twisted-nematic liquid crystal display which uses metal gratings for polarization of light as well as for liquid crystal alignment was fabricated.

Supervisor: Dr. Henry I. Smith
Adjunct Professor of Electrical Engineering

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